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WHISENANT,
S.G.

Triclopyr Residues and Persistence in Forest Vegetation, Litter, and Soil

Coop agreement
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Brigham Young University
Department of Botany and Range Science

FINAL REPORT FOR COOPERATIVE AGREEMENT
No. 22-C-6-INT-048
with BRIGHAM YOUNG UNIVERSITY
FS Contact: E. Durant McArthur
Cooperator Contact: Steve Whisenant

10 May 1988

Chief, USDA, Forest Service
ATTENTION: Director of FPM
P.O. Box 90609
Washington D.C. 20013-6090

Dear Sirs:

Enclosed is the final Progress Report for the NAPIAP study entitled "Triclopyr Residues and Persistence in Forest Vegetation, Litter, and Soil". We plan to submit a manuscript on this study to the *Bulletin of Environmental Contamination and Toxicology*. We will send reprints of that publication when available.

Please contact me if you have any questions.

Sincerely,

Steve Whisenant

Steven G. Whisenant
Associate Professor

401 WIDB Brigham Young University Provo, Utah 84602 (801) 378-2582

TITLE: Triclopyr Residues and Persistence in Forest Vegetation, Litter, and Soil

Final Progress Report

PRINCIPAL INVESTIGATOR:
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CO-INVESTIGATOR:
Dr. E. Durant McArthur

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May 10, 1988

DATES COVERED: April 1, 1986 to February 29, 1988.

SUBMITTED TO:
Chief, USDA Forest Service, Attention: Director of FPM, P.O. Box 96090, Washington D.C. 20013-6090.

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SUMMARY

Triclopyr (3,5,6-trichloro-2-pyridinloxyacetic acid) is a selective hormone-like herbicide which is becoming an important woody plant control measure in inland Pacific Northwest forests. Despite this increasing use of triclopyr, no information is available on the persistence of triclopyr in inland Pacific Northwest forests. This study describes the residue persistence of triclopyr at two conifer release sites in northern Idaho. Triclopyr residues in the vegetation, litter, and soil were examined for 12 months following application of 2.3 kg ae of the butoxyethyl ester of triclopyr ha^{-1}. Triclopyr disappearance in the foliage of all species combined was best explained by the nonlinear equation: $Y = 242(\text{number of days after application})^{-0.696}$, where Y is the triclopyr concentration in $\mu g\ g^{-1}$, ($r^2=0.69$). Triclopyr concentration in the vegetation varied among species at both sites. One day after application, the highest concentration, 362 $\mu g\ g^{-1}$, occurred in shinyleaf ceanothus leaves. Although shinyleaf ceanothus is not palatable to large herbivores, a 60 kg whitetail deer can eat 5.0 kg shinyleaf ceanothus leaves per day with no effect, based on the highest residues, which only last a few days. Based on the average for all species at day 1, a 42% decline in triclopyr concentration occurred after 6 days, 72% in 28 days, and more than 98% 365 days after application. The soil at one of the sites had a mineral soil-surface while the other had a very thick shrub cover and a leaf-litter layer with an underlying O horizon. The site with the bare soil had the highest triclopyr residues immediately after the herbicide application. The site with a heavy shrub cover had the highest residue concentrations when the shrubs began to defoliate as a result of the herbicide.

INTRODUCTION

Triclopyr (3,5,6-trichloro-2-pyridinyloxyacetic acid) is a relatively new, selective hormone-like herbicide somewhat similar to the phenoxy herbicides and picloram (4-amino-3,5,6-trichloro-2-pyridinecarboxylic acid) in structure and activity (Bovey and Meyer 1981). Triclopyr effectively controls many woody plants and is increasingly important for woody plant control in the Pacific Northwest (Boyd *et al.* 1985). Current registration in the Pacific Northwest is for broadcast applications of the ester formulation (Garlon 4) for control of unwanted woody plants and broadleaf weeds in forest site-preparation and conifer release. Non-crop uses include woody plant control on rights-of-ways such as electrical power lines, communication lines, pipelines, roadsides and railroads, and fence rows.

Recommended spray volumes for forest applications are 50 to 235 liters ha^{-1} by air and 94 to 940 liters ha^{-1} with ground applications. Triclopyr (1.1 to 2.5 kg ha^{-1}) may be used on conifer plantations (excluding pines) after conifers harden off in late summer and while hardwoods are still actively growing. Treatment should occur as soon after conifer bud hardening as possible so that hardwoods are actively growing. Douglas fir [*Pseudotsuga menziesii* var. *glauca* (Beissn.) Franco] plantations in northern Idaho are usually sprayed in early August.

Triclopyr has demonstrated promise as an effective control measure on several undesirable forest and range plants (Bovey *et al.* 1981, Boyd *et al.* 1985). Current registration allows the use of triclopyr in site preparation, conifer release, and vegetation control on rights-of-way in the Pacific Northwest. Norris *et al.* (1987) determined the movement and persistence of triclopyr in grass, soil, and water of a western Oregon hill-pasture. However, no published information on the persistence of these herbicides in forest soils or vegetation of the inland Pacific Northwest was found.

The intake—by terrestrial animals—of significant amounts of herbicide is most likely to result from consumption of recently treated vegetation (Norris 1977). Norris *et al.* (1977) found that the average amount of herbicide, when measured immediately after application of 2.24 kg 2,4,5-T ha^{-1} was about 50 $\mu g\ g^{-1}$ in the foliage, with much less present on the forest floor or in the soil. Herbicides disappear rapidly due to processes such as volatilization, runoff, leaching, degradation, weathering, absorption, growth dilution, metabolism, excretion, and

adsorption (Norris 1981). The dissipation of herbicides, the typically reduced palatability of the dead vegetation, and the physical and preferentially limits on consumption should prevent significant chronic exposure of phenoxy herbicides (Norris 1977).

Objectives. The objectives of this research were to determine residue persistence of triclopyr at two conifer-release sites in northern Idaho. Triclopyr residues in the vegetation, litter, and soil were examined for 12 months after application of 2.3 kg ae of the butoxyethyl ester of triclopyr ha^{-1}. Data collected in this research should enable resource managers to better evaluate potential problems resulting from triclopyr residues in vegetation, leaf-litter, and soil.

METHODS AND EXPERIMENTAL DESIGN

Study sites. Two sites near Bovill, Idaho[1] were selected for treatment with triclopyr (butoxyethyl ester). Four 9.1- by 23-m plots were marked at each site. Application were made along transects in the center of each plot.

Site 1 had a southwest exposure averaging 31% slope, and was dominated by shinyleaf ceanothus (*Ceanothus velutinus* Dougl. ex. Hook.) (88% cover with plants averaging 0.96 m high). The soil on this site was a silt (medial over loamy-skeletal mixed, frigid Typic Vitrandpt). Chemical and physical characteristics of the mineral soil are in Table 1. Douglas fir plants averaged 0.48 m high. Approximately 9% of the Douglas fir plants on this site had been recently killed. The lack of root damage on dead plants indicated the mortality may have been caused by being overtopped by shinyleaf ceanothus rather than from rodent damage. The Free to Grow (FTG) rating for this site was 4 (significant competition overtopping crop tree; survival is questionable).

Site 2 has a northeast exposure averaging 10% slope, with a silt loam soil (medial over loamy-skeletal mixed, frigid Typic Vitrandpt). The soil surface was mostly uncovered and did not have an organic layer. Chemical and physical characteristics of the mineral soil are in Table 1. Only approximately 9% of the ground was covered with shinyleaf ceanothus and few Douglas fir plants were overtopped. The Free to Grow (FTG) rating for this site was 1 (free to grow

[1]The authors acknowledge the assistance of Dan Miller and the Potlatch Corporation, Bovill, Idaho. His assistance in locating the study area and in providing precipitation data is greatly appreciated.

without significant competition). This site had several additional species, such as Wood's rose (*Rosa woodsii* Lindl), sticky currant (*Ribes viscosissimum* Pursh), mountain lover [*Pachistima myrsinites* (Pursh) Raf.], and snowberry [*Symphoricarpos albus* (L.) Blake].

Triclopyr applications. The butoxyethyl ester of triclopyr (2.3 kg ae ha^{-1} with 1.2 L polyglycol surfactant, 2.3 L ha^{-1} of diesel oil, and enough water to give 93.5 L ha^{-1} total solution) was applied to both study sites on 10 August 1986. Site 1 was treated at 24C and 38% relative humidity. Site 2 was treated at 28C and 31% relative humidity. The herbicide mixture was applied with a compressed-air powered, backpack sprayer. The sprayer had a single nozzle at 3.2 m above ground and a 3.1 m swath width.

Experimental design and analysis. The study was designed as a completely randomized experiment. The data were handled in separate analyses as follows: (1) residues in vegetation handled as a factorial with species and time after application as the factors, these data were analyzed by site since one site had more species than the other site, (2) residues in the litter were analyzed as a completely random experiment with time after application as the factor, and (3) residues in soils were analyzed as a factorial experiment with site, time after application and depth in the soil profile as the factors.

Sampling procedures. Soil, vegetation and leaf-litter samples were collected at 1, 6, 28, 76, 277, and 365 days after application of triclopyr. Samples were frozen after collection and kept frozen until extraction and analysis. Foliage and the terminal 15 cm of woody plant twigs were collected. Grass foliage samples were clipped to ground level. These vegetation samples represent the most likely sources of food for large herbivores. Leaf-litter was collected from two 15- by 15-cm areas from each plot. Soil samples were collected from 0 to 5, 5 to 15, and 15 to 30 cm depths. New soil pits were made at each sampling date. Tools used for collecting soil samples were cleaned with acetone after each sample collection to prevent contamination. Vegetation, leaf-litter or soil samples were collected in duplicate at each of the five plots per site. Composite samples of vegetation, leaf-litter or soil were prepared in the laboratory by combining equal amounts of material from each of the two subsamples from a given plot.

Cumulative precipitation, soil organic matter, clay content, time after application and soil pH were used as independent variables in a stepwise multiple regression analysis to predict concentration, movement, and degradation of triclopyr in the soil. Triclopyr residue concentrations in the soil and litter were used as dependent variables with time, soil depth, and cumulative precipitation being the independent variables.

Analytical techniques. Triclopyr was extracted from vegetation and soil samples by blending the leaf material or soil in a 7:1 mixture of acidified acetone:water for 3 minutes. The mixture was then filtered and reduced in volume prior to adjusting pH to 8.0–8.5 with $NaHCO_3$. The triclopyr was next extracted from the acetone with 2 liquid-liquid separations in hexane. The mixture was next reduced in volume and methylated with BF_3-methanol prior to analysis with a gas chromatograph. Triclopyr recovery averaged 89% for Douglas fir, 87% for shinyleaf ceanothus, 92% for Wood's rose, 93% for grasses, 90% for mountain lover, and 94% for snowberry at 0.1 $\mu g\ g^{-1}$. The recovery from fortified soils averaged 86.7% triclopyr at 0.1 $\mu g\ g^{-1}$.

RESULTS

Vegetative material. Great differences were found in initial residue levels in the vegetation samples. Norris *et al.* (1977) also found big differences in herbicide concentrations of plant foliage following applications of 2,4,5-T. Shinyleaf ceanothus terminal branch segments had the highest triclopyr concentrations, possibly because its broad leaves intercepted more spray material.

Triclopyr concentrations in the vegetation varied among species at both sites. One day after application, the highest concentration, 362 $\mu g\ g^{-1}$, occurred in shinyleaf ceanothus on the southwest slope (Table 2). Based on the average for all species at day 1, a 42% decline occurred after 6 days, 72% in 28 days, and more than 98% 365 days after application. The lowest initial concentration occurred in Wood's rose on the northeast slope (Table 3).

Wood's rose, sticky current, and snowberry were killed by the herbicide application. Thus, no foliage of those species was available after the 28 day collection period. Shinyleaf ceanothus samples collected 277 and 365 days after treatment contained only dead leaves which had not dropped.

Mean herbicide concentrations were markedly different among species and sampling time. There was a highly significant species by sampling time interaction at each site, indicating the rate of change of triclopyr concentration was not the same among species. Leaves which had died and dropped from the plant were not included in the sampled vegetation. However, triclopyr concentrations in dropped leaves was reflected in the leaf-litter samples. Vegetation retained on the plants was sampled because it was considered to be most likely to be consumed by large herbivores.

The change in triclopyr concentration in all species (combined) was best explained by the nonlinear equation: $Y = 242(\text{days after application})^{-0.696}$, where Y is the triclopyr concentration in $\mu g\ g^{-1}$, ($r^2=0.69$). Regression equations describing triclopyr disappearance in each species are found in Table 5. Regression equations for triclopyr disappearance in the individual species explained 83 to 97% of the variation using a single independent variable, time (days) after application (Table 5).

Residue levels observed at a specific point in time are the result of a number of processes, all of which may proceed at different rates. Processes such as volatilization, rain washing and translocation may predominate shortly after application, while weathering, root exudation and metabolism may be more important later (Norris *et al.* 1984).

No observable effect level (NOEL). The chronic or subchronic oral NOEL is the maximum daily oral dose of herbicide which does not exceed the no observable effect level (e.g. for toxicity, fetotoxicity, teratogenicity, oncogenicity, or mutagenicity) when administered orally over a relatively long period of time or during a critical time period (e.g. pregnancy). The NOEL may be considered as the highest dose which produces no response and is expressed as mg of herbicide active ingredient per kg of body weight per day. This is a much more conservative measure of effect than is the more commonly used LD_{50} (lethal dose to 50% of treated animals).

Triclopyr's NOEL value (30 mg triclopyr kg^{-1} forage day^{-1}) is only available for rats and rabbits (Weed Science Society of America 1983). Although imperfect as a measure of susceptibility to other animals, this value can be extrapolated for

larger animals. A hypothetical amount of forage that can be ingested without exceeding the NOEL can be calculated with the following formula:

$$X = W \times T / C$$

where X is the hypothetical amount of forage (kg) that can be ingested without exceeding the NOEL (daily dose); W is the body weight of the animal involved (kg); T is the NOEL (30 mg triclopyr kg^{-1} of animal weight); and C is the concentration of triclopyr acid equivalent in the leaf material.

The highest triclopyr concentration (362.6 mg kg^{-1}) was found on shinyleaf ceanothus foliage, 1 day after application. Using this number in the formula for a 60 kg whitetail deer indicates that a 60 kg whitetail deer can eat 5.0 kg ceanothus leaves per day with no effect. This 'worst case scenario' is very unlikely to occur since shinyleaf ceanothus is not a highly preferred forage to most large herbivores. A diet study conducted in the Chopaka Mountains in Washington (Campbell and Johnson 1983) indicated that during the summer shinyleaf ceanothus comprised 3.1 and 0.6% of the diets of mule deer and cattle (on a relative percent density of plant fragments in fecal samples).

Leaf-litter. There was little leaf-litter present on the northeast site, so leaf-litter samples were collected at the southwest site only. One day after spraying the average triclopyr concentration in leaf-litter was 0.01 $\mu g\ g^{-1}$, but the variability was high because of interception by the shinyleaf ceanothus canopy (Table 2). Shinyleaf ceanothus covered 88% of the soil surface at the southwest site. The canopy intercepted most of the triclopyr before it reached the soil surface. The low August precipitation probably indicates little of the triclopyr was rinsed off the leaves onto the soil (Fig 1). Having the triclopyr on the leaf surface for a long time period may have resulted in a high percentage of the applied triclopyr being absorbed into the leaf (or volatilized into the atmosphere). As a result, little of the herbicide reached the litter layer until leaves begin dropping.

Triclopyr concentrations in the leaf-litter increased dramatically between 28 and 76 days after application. This probably reflects increased precipitation and some leaf drop from leaves in the upper canopy. Leaf-litter triclopyr concentrations dropped dramatically between 277 and 365 days after application. Summer temperatures combined with snow-melt water apparently combined to greatly increase triclopyr disappearance from leaf-litter. No triclopyr was detected in leaf-litter samples 365 days after application.

Soil. The northeast site had no organic matter accumulations on the soil surface, thus leaving the mineral soil exposed to the herbicide application. The southwest exposure site was largely covered by shinyleaf ceanothus (88% cover), had a solid leaf-litter cover, and an organic matter layer. The mineral soil on the northeast site was directly exposed to the triclopyr spray. Thus, triclopyr concentrations in that soil were much higher than that of the southwest site (Table 4). Triclopyr concentrations on the northeast site decreased steadily until winter (between the 76 and 277 day samples) arrived. Then decreased rapidly during the summer. At the southwest site, triclopyr did not appear in the soil until the 28 day sampling period. Triclopyr residues in the soil of the southwest site peaked at 76 days for the O horizon and 277 days for the mineral soil (Table 4).

Schubert *et al.* (1980) reported that triclopyr residues decreased from 18.0 to 0.8 mg kg^{-1} in 28 days and to 0.1 mg kg^{-1} in 166 days in a West Virginia watershed. In a Texas field study on a Miller clay loam soil, Moseman and Merkle (1972) found that triclopyr applied in fall persisted about 6 months, whereas most of that applied in summer was dissipated in 3 months. Norris *et al.* (1987) found that triclopyr disappeared from soil more slowly than has be indicated in other studies. Triclopyr residues in soil decreased with time, following first-order kinetics. The more rapid disappearance in West Virginia (Schubert *et al.* 1980) and the Texas summer data (Moseman and Merkle 1972) probably reflects their relatively wet summers in contrast to the dry Oregon summer. In the current study, the rate of triclopyr disappearance greatly decreased during the winter. The soils in this northern Idaho study were under a snow cover for most of the winter. This greatly reduced the triclopyr disappearance rate.

CONCLUSIONS

Triclopyr concentrations varied significantly between species, sites, and sampling dates. The highest concentrations were found in shinyleaf ceanothus leaves. Wood's rose, sticky current, and snowberry were killed by the triclopyr application; thus no foliage was available after the 28th day collection. Shinyleaf ceanothus samples collected 277 and 365 days after treatment contained only dead leaves which had not dropped.

Although shinyleaf ceanothus is not highly palatable to the large herbivores of the region a 60 kg whitetail deer can eat about 5.0 kg ceanothus leaves per day with no effect. Cattle and elk would have to eat proportionally more of the treated foliage (based on body weight). These calculations are based on triclopyr residues which only last for a few days after application.

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Table 1. Chemical and physical characteristics of mineral soils on study sites near Bovill, Idaho in August 1986.

Site	Depth (cm)	Textural Components			Organic Matter (%)	pH
		Sand (%)	Silt (%)	Clay (%)		
Northeast						
	0-5	36.7	48.1	15.2	8.9 ± 1.8	6.5
	5-15	40.0	45.8	14.2	6.1 ± 0.1	6.0
	15-30	40.6	44.5	14.9	4.7 ± 0.7	6.2
Southwest						
	0-5	34.1	51.4	14.5	11.8 ± 2.0	6.3
	5-15	35.1	52.4	12.5	6.5 ± 0.2	6.1
	15-30	41.6	43.4	15.0	4.9 ± 0.6	6.1

Table 2. Triclopyr levels in leaf litter and terminal branch segments following application of 2.3 kg triclopyr ha^{-1} to a southwest slope near Bovill, Idaho.

Species	Days after Applications					
	1	6	28	76	277	365
	($ug\ g^{-1}$)					
Leaf litter	0.1 ± 0.1	0.1 ± 0.1	0.2 ± 0.1	0.4 ± 0.3	0.3 ± 0.4	0.0 ± 0.0
Shinyleaf ceanothus	362.6 ± 243.9	253.7 ± 106.8	96.2 ± 8.6	46.1 ± 7.0	24.5 ± 8.6	4.7 ± 0.7
Douglas fir	198.9 ± 160.5	81.5 ± 13.4	9.4 ± 3.4	7.6 ± 2.6	3.3 ± 1.5	1.2 ± 0.8

Table 3. Triclopyr levels in terminal branch segments following application of 2.3 kg triclopyr ha^{-1} to a northeast slope near Bovill, ID.

Species	Days after application					
	1	6	28	76	277	365
	----------(ug g^{-1})----------					
Shinyleaf ceanothus	354.7 ± 207.2	249.9 ± 32.6	187.0 ± 41.3	74.1 ± 20.0	25.7 ± 5.8	6.7 ± 1.1
Douglas fir	151.4 ± 29.5	58.0 ± 36.8	20.3 ± 7.0	6.7 ± 1.8	2.4 ± 0.8	1.5 ± 0.4
Mountain lover	176.3 ± 70.3	75.0 ± 40.6	32.3 ± 4.4	15.3 ± 8.1	7.6 ± 3.0	2.6 ± 0.8
Woods rose	79.1 ± 21.0	49.7 ± 2.8	33.2 ± 3.1	-----	-----	-----
Sticky currant	100.4 ± 23.6	68.6 ± 11.8	35.7 ± 1.3	-----	-----	-----
Snowberry	108.6 ± 19.9	81.5 ± 7.7	49.9 ± 18.9	-----	-----	-----
Grasses	156.7 ± 45.2	53.6 ± 14.7	11.4 ± 4.0	4.7 ± 1.3	1.2 ± 0.5	0.2 ± 0.1

Table 4. Triclopyr levels in soil following application of 2.3 kg triclopyr ha^{-1} near Bovill, Idaho on 10 August 1986.

Site	Soil layer	Days after application					
		1	6	28	76	277	365
		----------(ug g^{-1})----------					
Northeast							
	Mineral soil 0-5 cm	1.62 ± 0.53	0.70 ± 0.44	0.65 ± 0.76	0.38 ± 0.15	0.32 ± 0.23	0.00 ± 0.00
	5-15 cm	0.00 ± 0.00	0.25 ± 0.37	0.26 ± 0.23	0.15 ± 0.15	0.18 ± 0.15	0.00 ± 0.00
	15-30 cm	0.00 ± 0.00	0.00 ± 0.00	0.04 ± 0.03	0.12 ± 0.11	0.12 ± 0.09	0.00 ± 0.00
Southwest							
	O horizon	0.00 ± 0.00	0.00 ± 0.00	0.34 ± 0.42	0.46 ± 0.47	0.33 ± 0.10	0.00 ± 0.00
	Mineral soil 0-5 cm	0.00 ± 0.00	0.00 ± 0.00	0.13 ± 0.18	0.12 ± 0.14	0.17 ± 0.17	0.00 ± 0.00
	5-15 cm	0.00 ± 0.00	0.00 ± 0.00	0.05 ± 0.04	0.05 ± 0.02	0.12 ± 0.18	0.00 ± 0.00
	15-30 cm	0.00 ± 0.00	0.00 ± 0.00	0.02 ± 0.03	0.03 ± 0.05	0.19 ± 0.17	0.04 ± 0.04

Table 5. Results of regression analyses of triclopyr residues in vegetation following application of 2.3 kg triclopyr ha^{-1} near Bovill, Idaho.

Species	Regression equations[1]	r^2
Shinyleaf ceanothus	$Y = 609\ X^{-0.626}$	0.83
Douglas fir	$Y = 217\ X^{-0.812}$	0.97
Mountain lover	$Y = 222\ X^{-0.655}$	0.95
Grasses	$Y = 257\ X^{-1.034}$	0.93
All vegetation	$Y = 242\ X^{-0.696}$	0.69

[1]Equations are of the form $Y = bX^a$, where Y is the triclopyr concentration in $\mu g\ g^{-1}$, b is a constant, X is the number of days after application and a is the exponent.

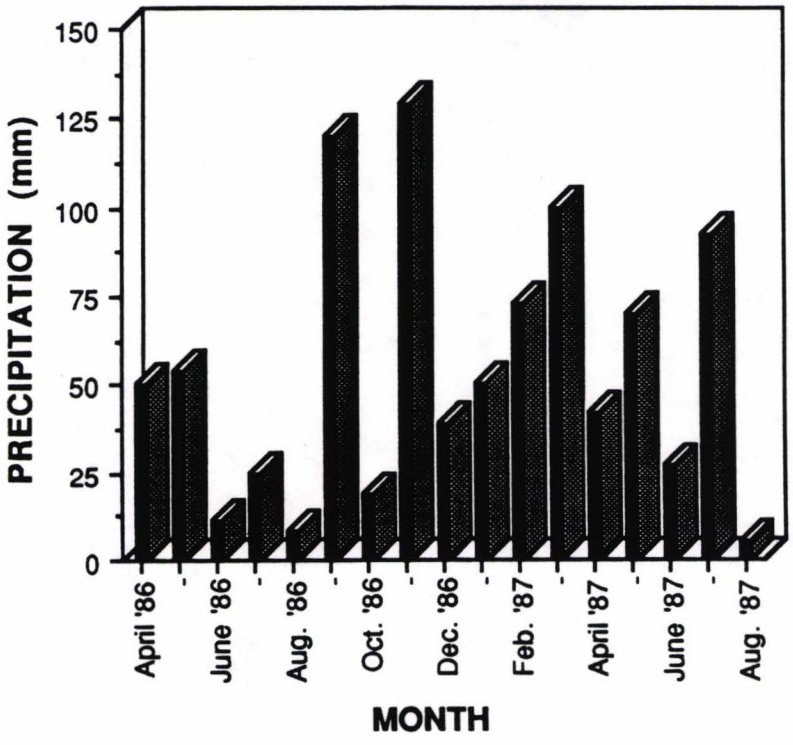

Fig. 1 Monthly precipitation in Bovill, Idaho from April 1986 through August 1987.

RECOMMENDATIONS FOR ADDITIONAL RESEARCH

Additional research designed to determine how much of these browse species is consumed by wildlife species would help evaluate the potential problems.

ACTUAL COSTS

	NAPIAP FUNDS[1]	BYU FUNDS
(A) SALARIES AND WAGES		
Principal Investigator	$ 7,415.00	$ 20,476.30
Students	2,397.00	
Non-Students	1,328.00	
Total Salaries and Wages	11,140.00	
(B) FRINGE BENEFITS	1,337.00	2,457.16
Total Salaries, Wages and Benefits	12,477.00	
(C) EQUIPMENT AND SUPPLIES	6,515.00	
(D) CAPITAL EQUIPMENT	2,678.00	
(E) TRAVEL	4,683.00	
(F) OTHER COSTS		
Publication Costs	0.00	
(G) TOTAL DIRECT COSTS (A, B, C, E, F)	26,353.00	22,933.46
(H) TOTAL INDIRECT COSTS		
Indirect Cost Recovery	0.00	9,632.05
Waived overhead	0.00	5,819.96
(I) TOTAL COSTS (D, G, H)	**$26,353.00**	**$38,385.47**
TOTAL	**$64,738.47**	

[2]Brigham Young University received these funds from the Intermountain Forest and Range Experiment Station as a Cooperative Agreement (USDA 22-C-4-INT-46).

PLANS FOR PUBLICATION

A manuscript entitled "Triclopyr Residue Persistence in Northern Idaho Browse Species" will be submitted to the *Bulletin of Environmental Contamination and Toxicology*. The manuscript will be submitted within 60 days of submission of this report. We will send reprints of the resulting manuscript to the USDA, Forest Service, Director FPM.

POTENTIAL PATENTS

No potential patents resulted from this research.

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Environmental Contamination and Toxicology

Triclopyr Persistence in Northern Idaho Forest Vegetation

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Triclopyr (3,5,6-trichloro-2-pyridinyloxyacetic acid) is increasingly becoming important for woody plant control (Boyd *et al*. 1985) in the inland Pacific Northwest. Current registration allows the use of triclopyr in site preparation, conifer release and vegetation control on rights-of-way. Norris *et al*. (1987) determined the movement and persistence of triclopyr in grass, soil, and water of a western Oregon hill-pasture, but no data are available on triclopyr persistence in the inland, Pacific Northwest.

The intake of significant amounts of herbicide, by terrestrial animals is most likely to result from consumption of recently treated vegetation (Norris 1977). Elk, deer, cattle, and horses may be exposed to triclopyr used for brush control along rights-of-way, fence rows, or in forested areas sprayed for conifer release or site preparation. Feeding studies in rats and mice established the no observable effect level at 30 mg kg^{-1} body wt (Weed Science Society of America 1983).

Our objectives were to determine persistence of triclopyr in the forage at two northern Idaho sites. Triclopyr residues in terminal twig and leaf segments were examined for 12 mon after application of 2.3 kg ae triclopyr ha^{-1}. Such information should be useful in determining the potential for poisoning and in determining if triclopyr may reasonably be used where elk, deer, cattle, and horses are present.

MATERIALS AND METHODS

Two sites near Bovill, Idaho were selected as study sites. Four 9.2- by 23-m plots were marked at each site. Site 1 had a southwest exposure averaging 31% slope and was dominated by shinyleaf ceanothus (*Ceanothus velutinus* Dougl. ex. Hook.) (88% cover with plants averaging 0.96 m high). The soil on this site was a silt (medial over loamy-skeletal mixed, frigid Typic Vitrandpt). Douglas fir [*Pseudotsuga menziesii* (Mirbel)Franco] plants averaged 0.48 m high.

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Site 2 had a northeast exposure averaging 10% slope, with a silt loam soil (medial over loamy-skeletal mixed, frigid Typic Vitrandpt). Only about 9% of the site was covered with shinyleaf ceanothus and few Douglas fir plants were overtopped. Site 2 had additional species, such as Wood's rose (*Rosa woodsii* Lindl), sticky currant (*Ribes viscosissimum* Pursh), mountain lover [*Pachistima myrsinites* (Pursh) Raf.] and snowberry [*Symphoricarpos albus* (L.) Blake].

The butoxyethyl ester of triclopyr (2.3 kg ae ha^{-1} with 1.2 L polyglycol surfactant, 2.3 L ha^{-1} of diesel oil, and enough water to give 93.5 L ha^{-1} total solution) was applied to four plots at both study sites on 10 August 1986. Site 1 was treated at 24C and 38% relative humidity. Site 2 was treated at 28C and 31% relative humidity. The herbicide mixture was applied with a backpack sprayer. The spray was applied with a single nozzle at 3.2 m above ground and a 9.2 m swath width.

The study was designed as a completely randomized experiment. Triclopyr residues were analyzed as a factorial with species and time after application as the factors, these data were analyzed by site.

Terminal branch and leaf samples were collected at 1, 6, 28, 76, 277, and 365 d after application of triclopyr. Samples were frozen after collection and kept frozen until extraction and analysis. Foliage and the terminal 15 cm of stems were collected. Grass foliage samples were clipped to ground level. These foliage samples represent the most likely sources of food for large herbivores. Two subsamples of each species were collected at each of the four plots per site. Composite foliage samples were prepared in the laboratory by combining equal amounts of material from the two subsamples.

Triclopyr was extracted from foliage samples by blending the leaf material in a 7:1 mixture of acidified acetone water for 3 minutes. The mixture was then filtered and reduced in volume prior to adjusting pH to 8.0–8.5 with $NaHCO_3$. The triclopyr was next extracted from the acetone with 2 liquid-liquid separations in hexane. The mixture was then reduced in volume and methylated with BF_3-methanol prior to analysis with a gas chromatograph by the McKellar method (McKellar, R. L. 1977. Determination of triclopyr; 3,5,6-trichloro-2-pyridinol and 2-methoxy-3,5,6-trichloropyridine in soil by gas chromatography. ACR 77.5. Dow Chemical Co. Midland, MI. 23 p.). Triclopyr recovery from fortified foliage samples averaged 89% for Douglas fir, 87% for shinyleaf ceanothus, 92% for Wood's

rose, 93% for grasses, 90% for mountain lover, and 94% for snowberry at 0.1 $\mu g\ g^{-1}$.

RESULTS AND DISCUSSION

Triclopyr concentrations in the foliage varied among species at both sites. One d after application, the highest concentration, 362 $\mu g\ g^{-1}$, occurred in shinyleaf ceanothus on the southwest slope (Table 1). Shinyleaf ceanothus foliage had the highest triclopyr concentrations, possibly because the broad leaves intercepted more spray material. Based on the average for all species at d 1, a 42% decline occurred after 6 d, 72% in 28 d, and more than 98% 365 d after application. The lowest initial concentration occurred in Wood's rose on the northeast slope (Table 1). Wood's rose, sticky current, and snowberry were killed by the herbicide application. Thus, no foliage of those species was available after the 28 d collection period. Shinyleaf ceanothus samples collected 277 and 365 d after treatment contained only dead leaves which had not dropped.

Mean herbicide concentrations were markedly different among species and sampling time. There was a highly significant species by sampling time interaction at each site, indicating the rate of change in triclopyr concentration differed among species. Dead leaves which dropped from the plant were not included in the samples.

The change in triclopyr concentration in all species (combined) was best explained by the equation: $Y = 242(\text{d after application})^{-0.696}$, where Y is the triclopyr concentration in mg g^{-1}, (r^2=0.69). Regression equations for triclopyr disappearance in the individual species explained 83 to 97% of the variation using a single independent variable, time (d) after application (Table 2). Residue levels observed at a specific point in time are the result of several processes, all of which may proceed at different rates. Processes such as volatilization, washing off from rainfall, and translocation may predominate shortly after application, while weathering, root exudation, and metabolism may be more important later (Norris *et al.* 1984).

A 450 kg cow that consumed 11.0 kg of shinyleaf ceanothus leaves (100% of total consumption) in the treated area, one d following application would receive about 8.8 mg triclopyr kg^{-1} body wt. A 60 kg whitetail deer in the treated area that consumed 1.5 kg of shinyleaf ceanothus leaves (100% of total consumption) one d following application would receive about 9.0 mg triclopyr kg^{-1} body wt. This

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Table 1. Triclopyr levels in terminal branch and leaf segments following application of 2.3 kg triclopyr ha^{-1} to a southwest slope near Bovill, Idaho.

Species	Days after Applications					
	1	6	28	76	277	365
	(µg g^{-1})					
	Southwest slope					
Shinyleaf ceanothus	362.6 ± 243.9	253.7 ± 106.8	96.2 ± 8.6	46.1 ± 7.0	24.5 ± 8.6	4.7 ± 0.7
Douglas fir	198.9 ± 160.5	81.5 ± 13.4	9.4 ± 3.4	7.6 ± 2.6	3.3 ± 1.5	1.2 ± 0.8
	Northeast slope					
Shinyleaf ceanothus	354.7 ± 207.2	249.9 ± 32.6	187.0 ± 41.3	74.1 ± 20.0	25.7 ± 5.8	6.7 ± 1.1
Douglas fir	151.4 ± 29.5	58.0 ± 36.8	20.3 ± 7.0	6.7 ± 1.8	2.4 ± 0.8	1.5 ± 0.4
Mountain lover	176.3 ± 70.3	75.0 ± 40.6	32.3 ± 4.4	15.3 ± 8.1	7.6 ± 3.0	2.6 ± 0.8
Woods rose	79.1 ± 21.0	49.7 ± 2.8	33.2 ± 3.1	-----	-----	-----
Sticky currant	100.4 ± 23.6	68.6 ± 11.8	35.7 ± 1.3	-----	-----	-----
Snowberry	108.6 ± 19.9	81.5 ± 7.7	49.9 ± 18.9	-----	-----	-----
Grasses	156.7 ± 45.2	53.6 ± 14.7	11.4 ± 4.0	4.7 ± 1.3	1.2 ± 0.5	0.2 ± 0.1

'worst-case' scenario is unlikely since shinyleaf ceanothus is not a desired forage species. A diet study conducted in the Chopaka Mountains of Washington (Campbell and Johnson 1983) indicated that during the summer, shinyleaf ceanothus comprised only 3.1 and 0.6% of mule deer and cattle diets, respectively.

Dosing cattle with 150 mg triclopyr kg^{-1} body wt resulted in mortality after 6 d dosing (Rowe 1980). No data are available on the "No Observable Effect Level" of cattle, deer, or elk. Ponies dosed at 60 mg kg^{-1} body wt had no significant clinical effects or lesions. For ponies, 300 mg kg^{-1} body wt appears to be an approximate multiple dose LD_{50}. The toxic usage in ponies was 5 times the estimated maximal intake for the highest recommended rate (Osweiler 1983). Triclopyr residue data from this study and large herbivore toxicological data from other studies indicate that poisoning from the proper use of triclopyr is unlikely.

Table 2. Results of regression analyses of triclopyr residues in foliage following application of 2.3 kg triclopyr ha^{-1} near Bovill, Idaho.

Species	Regression equations[1]	r^2
Shinyleaf ceanothus	$Y = 609\ X^{-0.626}$	0.83
Douglas fir	$Y = 217\ X^{-0.812}$	0.97
Mountain lover	$Y = 222\ X^{-0.655}$	0.95
Grasses	$Y = 257\ X^{-1.034}$	0.93
All foliage	$Y = 242\ X^{-0.696}$	0.69

[1]Equations are of the form $\underline{Y} = b\underline{X}^{\underline{a}}$, where $\underline{Y}$ is the triclopyr concentration in $\mu g\ g^{-1}$, $\underline{b}$ is a constant, $\underline{X}$ is the no. d after application and $\underline{a}$ is the exponent.

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